

SHELL CHEMICAL YABUCOA INC.

Attachment II-1

Waste Analysis Plan

WASTE ANALYSIS PLAN

**Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico**

May 2002

Shell Chemical Yabucoa, Inc. (SCYI) has developed this Waste Analysis Plan (Plan) to satisfy the requirements of 40 CFR 264.13(b). The objective of this Plan is to establish procedures to be implemented by SCYI to obtain information needed to successfully store hazardous waste at its Hazardous Waste Storage Area (HWSA). Information gathered as a result of this Plan will be used by SCYI personnel to ensure that wastes will be managed in a manner that is protective of human health and the environment.

This Plan applies only to those hazardous wastes which are generated on-site and are stored in SCYI's regulated HWSA. SCYI does not accept waste from any off-site source.

As described in Chapter 1 of the RCRA Part B Permit Application, a comprehensive characterization of the hazardous wastes generated at the SCYI facility has been completed. This Plan is designed to ensure that initial waste classification is accurate and is up-to-date. The Plan will be modified as required to conform to changes in facility processes that may impact waste composition and to revisions in applicable hazardous waste regulations.

This Plan was developed in accordance with applicable USEPA guidance, including "Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, A Guidance Manual," OSWER 9938.4-03, April 1994.

I. FACILITY DESCRIPTION

This section provides a brief description of SCYT's manufacturing process, waste generation activities, and hazardous waste management unit. Detailed information on each of these topics can be found elsewhere in SCYT's Part B Permit Application.

A. Facility Processes and Activities

The SCYT facility is a petroleum refinery which historically processed 85,000 barrels per day (bpd) of crude oil. Major products included: reformate, kerosene, light distillates, white oil, naphtha, jet fuel, diesel fuel, No. 2 fuel oil, desulfurized gas oil, lube oil base stocks, residual fuels, aromatic extracts, slack wax and sulfur. The facility operates continuously 24 hours a day, seven days a week, in three shifts. Due to recent reconfiguration in company operations, the facility is operating at a reduced output and scale. These changes will continue for the foreseeable future.

Operations at the SCYT facility are conducted at the three distinct areas - the Refinery Area, the Tank Farm and the Dock Area. The three areas are interconnected by a series of above ground pipelines. Further information on each of the areas is provided below.

Refinery Area - The Refinery Area includes SCYT's oil processing operations. The following process units are located at the Refinery Area: crude unit, gas oil desulfurizer, hydrogen unit, sulfur unit, hydrotreater and feed preparation unit, MEK dewaxing unit, UV stabilization and final distillation unit, gasoline reformer and utilities. The Refinery Area also includes tank storage facilities, administrative and maintenance operations and a waste storage and treatment facility. The Refinery Area Wastewater Treatment Plant is located in the northern portion of the Refinery Area.

Tank Farm - Raw materials and products manufactured by SCYT are stored in above ground steel tanks in the Tank Farm Area. Materials are conveyed to and from the Tank Farm via above ground pipelines. The Tank Farm contains approximately 43 crude and product tanks, ranging in size from 500 to 375,000 barrels. In addition, there are three storage tanks associated with SCYT's ballast water and slop oil management systems. A tanker truck loading rack is located near the southwestern corner of the Tank Farm. The

Tank Farm Wastewater Treatment Plant is located in the southeast corner of the Tank Farm.

Dock Facility - SCYI operates a dock facility for the loading and unloading of crude oil and products. The facility includes a Main Dock which serves oil tankers and a Barge Dock which serves smaller vessels and barges. There is also a dock for the servicing of tugboats. Crude oil and products are transferred to and from the Dock Area via aboveground pipelines. There are no storage facilities, process units or any other operations conducted at the Dock Area.

The SCYI facility is served by several different wastewater collection and treatment systems. The systems are designed to reduce the volume of contaminated wastewater requiring treatment by segregating contaminated and uncontaminated wastewater to the maximum extent possible. At the Refinery Area, collection systems exist for contaminated process wastewater, contaminated storm water, uncontaminated storm water, and sanitary wastewater. At the Tank Farm, a collection system is provided for storm water and tank water drawoff. Collection systems are provided for storm water at both the Main Dock and the Barge Dock.

B. Classification and Quantities of Hazardous Waste

SCYI generates a number of hazardous wastes subject to Subtitle C of RCRA. These wastes are managed in full compliance with applicable sections of 40 CFR Parts 260 through 270 and equivalent Commonwealth of Puerto Rico regulations. At present, the facility operates a single RCRA-regulated hazardous waste management unit. This unit, the Hazardous Waste Storage Area (HWSA), is used for the storage of hazardous waste generated throughout the site prior to off-site disposal. A full description of the unit is provided below in Section C.

Approximately 98% of the hazardous waste routinely generated at the SCYI facility and transported off-site for treatment and/or disposal is sludge from the process sewer, the wastewater treatment plant, slop oil tanks or bulk storage tanks. Historically, this has totaled approximately 675 tons per year. Generally, these wastes are dewatered using a plate filter press in order to reduce the volume of sludge requiring off-site treatment and/or disposal. Dewatered sludge is placed in suitable containers and transferred to SCYT's Hazardous Waste Storage Area prior to off-site shipment. Further description of hazardous wastes generated at the SCYI facility is presented below.

F037 - Petroleum Refinery Primary Oil/Water/Solids Separation Sludge - This waste is generated from refinery process sewers, wastewater treatment tanks (W5 and W6), oil recycling system tanks (103, 10 and W1), and process waste units located upstream of API separators. The material is not subject to regulation until it is removed from the sewer, tanks or units. The waste is generated on a periodic basis during routine maintenance and repair activities. The pumpable portion of the sludge is pumped into a vacuum truck while the hardened sludge is removed by shovels. Sometimes, water is added to loosen the sludge and make it pumpable. The dry hardened sludge is stored in 55 gallon drums at the HWSA. The pumpable wet sludge is dewatered and stored in roll-off containers at the HWSA.

F038 - Petroleum Refinery Secondary Oil/Water/Solids Separation Sludge - This waste is generated at units where secondary oil/water/solids separation occurs and at certain downstream units. Wastes include sludge from the DAF and IAF Units as well as IAF float. F038 sludge is generated on a periodic basis when maintenance work is performed in these units. The pumpable portion of the sludge is pumped into a vacuum truck while the hardened sludge is removed by shovels. Sometimes, water is added to loosen the sludge and make it pumpable. The pumpable sludge is dewatered and stored in roll-off containers at the HWSA before being shipped off-site for disposal. The dry hardened sludge, shoveled from the units, is containerized for disposal. Float from the IAF Unit is continuously generated and is conveyed to the Watery Oil Separator. The material is ultimately recovered in the Crude Unit.

K048 - Dissolved Air Flotation (DAF) Unit Float - The DAF Unit provides process wastewater with secondary oil/water/solids separation. Float from that DAF Unit is continuously generated and is conveyed to SCYT's slop oil tanks W5 and W6. The material is ultimately recovered in the Crude Unit.

K049 - Slop Oil Emulsion Solids - SCYI operates four Slop Oil Tanks - Tanks 103, W5 and W6 in the Refinery Area and Tank 10 in the Tank Farm. The slop oil tanks are routinely taken out of service at which time they are cleaned, inspected and, if necessary, repaired. Recovered oil is processed in the Crude Unit. The water phase, which may contain emulsion solids, is discharged into the process sewer. The solids ultimately settle in the API separator and, upon removal, are managed as API separator sludge (K051).

K050 - Heat Exchanger Bundle Solids - Refinery Area heat exchanger bundles are periodically cleaned using steam cleaning, high pressure hydroblasting or other suitable means. Wash water and solids generated during the process are discharged by a drainage system to the process sewer system, which flows into the 2-Cell API Separator. Solids settle in the API separator and, upon removal, are managed as API separator sludge (K051). Heavy solids on the concrete pad, which are not captured by the drainage system, are shoveled into drums. The drummed material is dewatered using a non-reactive sorbent material to remove any free liquid and is managed as K050.

K051 - API Separator Sludge - Solids are periodically removed from the Refinery 2-Cell and 3-Cell API Separators as well as the Tank Farm East and West API Separators. These solids are dewatered, containerized in roll-off containers and transported off-site for disposal.

K169 - Crude Oil Tank Sediment - SCYI may periodically generate sediment when cleaning out crude oil tanks. This sediment will be dewatered using non-reactive sorbent material or other suitable means, containerized in roll-off containers and transported off-site for disposal.

K171 - Spent Hydrotreating Catalyst - During process unit turnarounds, approximately once every three to five years, SCYI generates a spent catalyst from the hydrotreater unit. The spent catalyst, which is a solid, is placed in lined 55-gallon drums upon removal from the process reactor. The catalyst is covered with a CO₂ blanket prior to closure of the container cover. Since this waste is listed by EPA due to its ignitable and toxic properties, it is stored in the area of the HWSA dedicated to storage of ignitable wastes prior to shipment to a reclamation facility for regeneration or reclamation.

K172 - Spent Hydrorefining Catalyst - During process unit turnarounds, approximately once every two years, SCYI generates a spent catalyst from the gas oil desulfurization (hydrorefining) unit. The spent catalyst, which is a solid, is placed in lined 55-gallon drums upon removal from the process reactor. The catalyst is covered with a CO₂ blanket prior to closure of the container cover. Since this waste is listed by EPA due to its ignitable and toxic properties, it is stored in the area of the HWSA dedicated to storage of ignitable wastes prior to shipment to a reclamation facility for regeneration or reclamation.

D001 - Ignitable Waste - Occasionally, small quantities of solid waste which may exhibit the characteristic of ignitability are generated. These materials may include spent solvents (not otherwise listed as hazardous waste), spill residues, off-specification product and miscellaneous laboratory and process waste. These wastes are classified as ignitable based on MSDS information, process knowledge and/or testing. These materials are placed in 55 gallon drums and stored in a designated area within the HWSA prior to off-site treatment, recovery or disposal.

D018 - Toxic (Benzene) - Process wastewater generated in the Refinery Area may contain benzene concentrations in excess of the TCLP levels. As a result, this material is classified as hazardous. D018 wastewaters are discharged into the 2-Cell API Separator. Primary separation is followed by dissolved air flotation in the DAF Unit and flow equalization in the Equalization Tanks. Testing has determined that the wastewater no longer exhibits the characteristic of toxicity after these initial treatment steps.

Tank bottoms from Reduced Crude Tanks and bottoms from the Desalter (process vessel) containing benzene are generated from routine maintenance and clean-out operations. Occasionally, small quantities of paint sludge is generated from routine painting operations using benzene thinner. These wastes may be classified as D018 due to the presence of benzene. The D018 waste is solidified with non-reactive sorbent material to remove free liquids and placed in 55-gallon drums.

U154 - Methanol - Off-specification methanol is periodically generated at the SCYI laboratory. This material is placed in suitable containers and is stored at the HWSA prior to off-site disposal. Methanol waste is managed as an ignitable waste.

Other Wastes - Other hazardous wastes may be periodically generated in limited quantities at the SCYI facility. These wastes may include laboratory waste, spill residues, excess or off-specification raw materials or products, etc. These wastes are placed in appropriate containers and are managed in full compliance with generator standards set forth at 40 CFR Part 262. The containers are stored at the HWSA for periods less than 90 days.

Hazardous constituents that may be present in the above wastes include petroleum hydrocarbons and metals. Table 1 presents a summary of the information relating to the basis for hazard classification. The above listed F- and K-wastes are classified by EPA as hazardous due to the potential presence of one or more of the following hazardous constituents: chromium, lead, arsenic, benzene, chrysene and benzo(a)pyrene.

C. Hazardous Waste Management Units

SCYI operates a single RCRA-regulated hazardous waste management unit - the Hazardous Waste Storage Area (HWSA). The HWSA is used by SCYI for the temporary storage of containers of hazardous waste prior to off-site shipment and disposal. Waste may be stored for periods exceeding 90 days. All containerized wastes are sealed to prevent accidental spillage.

After completion of the proposed upgrade, the HWSA will consist of two adjacent structures: an enclosed storage building and a covered concrete slab. The total storage capacity of the HWSA will be 717 cubic yards (cy) of hazardous waste. The total area of the unit will be approximately 6,810 square feet (sf).

The enclosed storage building includes a 4-inch thick, reinforced concrete floor, a galvanized sheet steel roof, steel siding on the north, south and east sides and a chain link fence with gate on the west side. The concrete floor is surrounded by an 8-inch high concrete block berm. Access to the building is via a ramp located on the west side of the building. The building is 60 ft long by 30 ft wide. The storage building is used for the storage of non-liquid hazardous waste in containers. Containers are placed on pallets and are transferred into and out of the building using a forklift. Containers are managed to minimize the potential for release of hazardous constituents within the building.

The proposed covered concrete slab area will consist of an 8-inch thick reinforced concrete slab surrounded by a continuous 4-inch high curb. The concrete slab will be used for the storage of liquid and non-liquid hazardous waste, primarily stored in roll-off dumpsters mounted on chassis and 55-gallon drums staged on pallets. The slab will be covered with a pre-fabricated, continuous span steel roof. The roof will not require supporting columns within the storage area. The storage area will be surrounded by a chain link fence with gates at access areas. Access to the storage area is via ramps located on the east, west and south sides of the building. The total area of the slab will be approximately 5,010 sf. The slab will be equipped with

trenches to drain spilled liquids and accumulated, wind-blown precipitation into blind sumps for removal.

II. SELECTION OF WASTE ANALYSIS PARAMETERS

Waste analysis parameters have been selected to provide SCYI with an accurate representation of the physical and chemical properties of the hazardous waste managed at the HWSA. The Plan will gather sufficient information to ensure:

- Compliance with applicable regulatory requirements
- Conformance with permit conditions
- Safe and effective waste management activities

A. Criteria and Rationale for Parameter Selection

SCYI considered the following criteria in identifying suitable waste analysis parameters to ensure safe and effective hazardous waste management activities:

Waste Identification - SCYI has relied on knowledge of the refining process and historical waste analysis data to properly identify and classify its hazardous waste. Due to the nature of SCYI's manufacturing processes, the physical and chemical characteristics of the hazardous wastes generated at the site are expected to remain relatively constant in the future. Stringent quality control requirements associated with the manufacture of petroleum products ensures that adequate information regarding chemicals used in the process is available. Testing standards and other quality assurance/quality control procedures are in place to ensure the quality and purity of each component within a process. No substance other than those specified may be introduced into a specific manufacturing process. Furthermore, as part of SCYI's site-wide Pollution Prevention Program, any proposed change to the manufacturing process must be evaluated by the Environmental Engineering Group for potential increases or changes in the generation, emission, or discharge of pollutants. Consequently, no hazardous constituent could be present in SCYI's waste stream without prior knowledge of its use in the manufacturing process.

SCYI's hazardous waste streams are either listed as hazardous under 40 CFR 261 Subpart D or exhibit a characteristic of hazardous waste as defined in 40 CFR 261 Subpart C. Routinely generated listed wastes include F037, F038, K049, K050, K051, K169, K171, K172 and U154 while the characteristic wastes include D001 and D018. SCYI applies process knowledge to identify and characterize listed hazardous sludges and catalysts routinely generated at the facility, including F037, F038, K049, K050, K051, K169, K171 and K172. Testing of these waste streams is not required to properly identify or characterize the wastes. Similarly, SCYI identifies and characterizes wastes resulting from discarded off-specification commercial chemical products of known physical and chemical constituents, such as the U-listed waste, by applying knowledge of the waste stream rather than testing. D001 wastes are identified and characterized as ignitable based on MSDS information, knowledge of the waste generating process or ignitability testing. Benzene-contaminated waste streams (D018) are identified and classified based on knowledge of the waste generating process or TCLP testing.

Identification of Incompatible/Inappropriate Waste - SCYI has not identified any wastes generated at the facility which are incompatible with other wastes or with the hazardous waste storage containers used by SCYI. Prior to introducing any new chemical to the manufacturing process, SCYI will assess the potential for incompatibility or reactivity problems and will make modification to its waste management system accordingly. Procedures for evaluating compatibility of materials are presented in Section VI.B of this Plan.

If SCYI has reason to suspect that a solid waste generated at the facility may exhibit a characteristic which would render it hazardous (i.e., ignitable, corrosive, reactive or toxic), the material will be classified as hazardous based on SCYI's knowledge of the chemical and physical properties of the material or on analytical results. If analytical results indicate that the waste is hazardous, or if SCYI makes such a determination without conducting sampling, the waste will be handled in full compliance with RCRA requirements. Application of process knowledge and waste analysis, if required, will ensure that hazardous waste remains properly classified in the future.

Based on consideration of the above criteria, SCYI has included in the Waste Analysis Plan a sampling and analysis program designed to ensure that the waste managed is in full

compliance with applicable RCRA requirements. Sampling and analysis of hazardous waste will be undertaken if any of the following conditions apply:

- The waste is of unknown physical or chemical characteristics or SCYI has reason to suspect that the waste exhibits ignitable, corrosive, reactive, or toxic characteristics, so as to render it hazardous.
- Changes in any manufacturing process may impact the characteristics of the hazardous waste and potentially increase risks to human health or the environment.
- Testing is required by the off-site TSDFs or reclamation facilities used to treat and/or dispose of the hazardous wastes generated at SCYI. In the event that a particular waste is rejected by a TSDF or reclamation facility, the waste will be sampled and analyzed as an unknown, heterogeneous waste in accordance with the procedures described in Sections IV (sampling) and V (analysis).

If a waste is subjected to sampling and analysis, the parameters selected will ensure proper identification and characterization of the waste. A list of parameters to be included in the sampling and analysis program is presented in Table 2 along with the rationale for selection. Sampling methods and frequency are discussed in Sections IV and V of this Plan, respectively. Application of SCYI's knowledge of chemicals used in the manufacturing processes along with analytical results will ensure that any changes in the physical or chemical characteristics of the waste are identified.

B. Special Parameter Selection Requirements

Waste analysis procedures for complying with "specialized" waste management regulatory requirements are incorporated into Table 2, as referenced above. A brief discussion on specialized waste management requirements is provided below.

Ignitable Waste - Waste being tested as per this Plan will be analyzed for ignitability if SCYI suspects the waste may exhibit the ignitable characteristic. As a safety precaution, all hazardous waste identified as ignitable is labeled and segregated at SCYI's HW SA. It is managed as ignitable waste until it is shipped off-site for proper disposal. Spent

pyrophoric catalysts (K171 and K172) are classified as hazardous waste due to toxicity and/or ignitability. As a safety precaution, upon removal from a process unit, these wastes are placed directly into 55-gallon drums, covered with a CO₂ blanket prior to closure of the container cover, and stored in the area of the HWSA dedicated to storage of ignitable wastes.

Reactive Waste - Although SCYI does not currently generate reactive waste, waste being tested as per this Plan will be analyzed for reactivity if SCYI suspects the waste may exhibit a reactive characteristic. As a safety precaution, any hazardous waste identified as reactive will be labeled and segregated at SCYT's HWSA. If necessary, SCYI will blanket the waste within a container with an inert substance such as CO₂. It will be managed as reactive waste until it is shipped off-site for proper disposal or reclamation.

III. SELECTION OF SAMPLING PROCEDURES

A. Sampling Methods and Equipment

Hazardous waste to be stored within the HWSA may be in the form of liquid, sludge or solid. Sampling procedures are set forth in SCYT's Standard Operating Procedure (SOP) No 001 entitled "Sampling of Hazardous Sludges and Solids" and SOP No. 002 entitled "Sampling of Hazardous Liquids." Copies of these SOPs are included in Attachment A to this Plan.

Sampling methods are summarized in Table 3. Specific sampling procedures differ depending upon the matrix of the waste, the type of container (e.g., 55-gallon drum vs. roll-off bin) and the homogeneity or heterogeneity of the waste. Procedures identified in the SOPs include, but are not limited to:

- Sample collection methodology;
- Number of samples;
- Decontamination procedures;
- Recordkeeping;
- Sample containers, preservation requirements and holding times;
- Chain of custody;
- Health and Safety requirements;
- Quality assurance/quality control.

B. Sample Preservation and Storage

Specific procedures for sample preservation and storage are provided in the SOPs described above.

C. Sampling QA/QC Procedures

Specific sampling QA/QC procedures are provided in the SOPs described above. QA/QC procedures are designed to satisfy the requirements set forth in Chapter 1 of "Test Methods For Evaluating Solid Waste: Physical/Chemical Methods," SW-846.

IV. LABORATORY TESTING AND ANALYTICAL METHODS

This section discusses how SCYI selects an analytical laboratory and selects testing and analytical methods for wastes subjected to sampling and analytical program.

A. Selection of Laboratory

All analyses associated with SCYI's waste analysis program are conducted by an independent laboratory. In selecting a specific laboratory, SCYI ensures that the laboratory has demonstrated experience and capabilities in the following areas:

Comprehensive QA/QC Program - The laboratory must document that a comprehensive QA/QC program is in place which is in full compliance with the requirements of Chapter 1 of "Test Methods For Evaluating Solid Waste: Physical/Chemical Methods," (SW-846). The QA/QC program must include qualitative QA/QC elements such as chain-of-custody protocol. The program must also use quantitative QA/QC measures including method blanks, duplicates, matrix spikes and surrogate spikes. Procedures to be followed by SCYI to monitor the laboratory to ensure all QA/QC objectives are met, are explained below.

Upon receiving each laboratory report, SCYI will review the case narrative to identify any QA/QC problems experienced by the laboratory. SCYI will check the chain of custody documentation for any breakage of sample containers, loss of sample integrity, use of improper sample containers, evidence of tampering and other problems. The continuity and completeness of sample tracking with chain of custody papers will be checked. The

report will be reviewed for inconsistencies in holding times and preservation techniques of samples.

On a quantitative basis, SCYI will review the report to ensure that the laboratory employed routine controls to evaluate the precision and accuracy of its analytical instrumentations by analyzing method blanks, duplicates, matrix spikes, surrogate spikes, and certified reference material. Results of the method blank analysis will indicate any contamination from analytical equipment or process. Similarly, analysis results of duplicates and certified reference material will evaluate the precision and accuracy of the analytical process respectively. Matrix spike results will determine the extraction efficiency of the method while the surrogate spike will indicate the effectiveness of the analytical process.

Technical Analytical Expertise - The laboratory selected must demonstrate proficiency in using established EPA analytical methods for hazardous waste determinations. The laboratory must demonstrate that analytical equipment is capable of consistently achieving required detection limits. To establish the laboratory's proficiency in using established EPA analytical methods, SCYI will hold a discussion with the laboratory's Project Manager on the knowledge of relevant methods, detection limits achievable for the analytes, versatility of the laboratory to adopt new EPA analytical methods, and certification of the laboratory (if applicable). SCYI will review the laboratory's QA/QC plan for consistency with EPA stipulated QA/QC procedures for appropriate methods. In addition, SCYI will review the data received by the laboratory for qualitative and quantitative elements of QA/QC as a routine check to determine the laboratory's proficiency in using established EPA analytical methods.

Information Management - The laboratory must maintain effective information management systems to assure that regulatory compliance can be verified and process performance can be evaluated.

B. Selection of Testing and Analytical Methods

The selection of analytical testing methods for the waste managed at SCYI's HWSA is based on the following four considerations:

- Physical state of the waste
- Analytes of interest
- Required detection limits

- Information requirements

Analytical methods selected are presented in Table 4. All methods are consistent with EPA-approved methods set forth in "Test Methods For Evaluating Solid Waste: Physical/Chemical Methods," (SW-846).

V. WASTE RE-EVALUATION

SCYT's waste re-evaluation program is designed to ensure that waste analysis will be repeated as required to ensure that waste classification is accurate and up-to-date. Waste re-evaluation analysis is conducted to identify any changes in the physical and chemical character of the waste managed at the HWSA Area. Waste re-evaluation will be performed as required based on the criteria described in Section II.A of this Plan, i.e., if a waste is of unknown characteristics, if SCYT suspects that a waste exhibits a hazardous characteristic, if SCYT changes any manufacturing process in a way that may significantly impact the characteristics of the hazardous waste generated, or if required by an off-site TSD facility or reclamation facility. Table 5 includes the list of parameters to be included in the sampling program for waste re-evaluation.

VI. SPECIAL PROCEDURAL REQUIREMENTS

A. Procedures for Receiving Wastes From Off-Site Generators

Since SCYT does not receive waste from off-site facilities, these requirements do not apply.

B. Procedures for Ignitable, Reactive and Incompatible Waste

Wastes managed at SCYT's HWSA may periodically exhibit the characteristic of ignitability based on knowledge of process or by ignitability testing, included as part of SCYT's waste analysis program. As a safety precaution, all hazardous waste identified as ignitable is labeled and segregated at SCYT's HWSA. It is managed as ignitable waste until it is shipped off-site for proper disposal.

SCYI routinely generates spent hydrotreating (K171) and hydrotreating (K172) catalysts which may exhibit a pyrophoric characteristic. SCYI manages these wastes as ignitable wastes since they are listed due to toxicity and ignitability. The material is stored in plastic-lined 55-gallon steel drums. An inert CO₂ blanket is placed over the material within the drum. The waste is properly labeled and segregated in the HWSA until shipment off-site for reclamation.

SCYI does not manage reactive wastes at the HWSA. However, if SCYI has reason to believe that a particular waste exhibits a reactive characteristic, that waste will be evaluated in accordance with the provisions of this Plan.

SCYI does not manage incompatible wastes at the facility. However, as a precaution, SCYI has developed procedures for evaluating new or uncharacterized wastes to ensure that any such wastes are properly managed in the future. These procedures are summarized below and are based on EPA's guidance manual entitled "A Method for Determining the Compatibility of Hazardous Waste" (EPA-600/2-80-076).

- Based on knowledge of the manufacturing process, identify all hazardous constituents expected to be present in the new or modified waste stream ("Waste A") which are not currently present in the waste managed at the HWSA ("Waste B").
- For each hazardous constituent identified, determine the "reactivity group number" (RGN) presented in Appendix I of the above-referenced guidance manual.
- Determine the RGN for each hazardous constituent present in Waste B.
- Using the Hazardous Waste Compatibility Matrix presented in Figure 1 of this Plan, identify the "reaction code" by cross referencing each constituent from Waste A with each constituent from Waste B.
- Verify that mixing of the constituents will not result in potential incompatible reactions.

SCYI will not manage any waste at the upgraded portion of the HWSA that is incompatible with the low permeability sealant. Compatibility of the waste with the sealant will be determined based on the adequacy of chemical resistance as provided in the manufacturer's specification.

C. Procedures to Ensure Compliance with LDR Requirements

All hazardous waste generated at SCYI is subject to land disposal restrictions (LDR) under RCRA. If SCYI intends to send a hazardous waste off-site to a RCRA landfill, testing will be conducted to ensure that the waste meets applicable treatment standards set forth at 40 CFR 268 Subpart D. LDR treatment standards for hazardous wastes generated at SCYI are summarized in Table 6. Sampling and analysis methods to be employed in determining compliance with the standards has been discussed in Section III and IV, respectively. If it is known that the wastes do not meet applicable LDR treatment standards based on process knowledge, and if the waste is not being sent to a landfill subject to LDR requirements, no testing will be conducted.

SCYI complies with all applicable LDR notification and certification requirements set forth at 40 CFR Part 268, as summarized below:

- If SCYI determines that a waste does not meet applicable LDR treatment standards, SCYI will notify the TSD facility in writing with each shipment. At a minimum, the notification will include the EPA hazardous waste number and the manifest number.
- If SCYI determines that a waste does meet applicable LDR treatment standards, SCYI will submit to the TSD a notice and certification with each shipment stating that the waste meets applicable LDR treatment standards. At a minimum, the notification will include the EPA hazardous waste number, the manifest number and waste analysis data if available. The certification will be signed by an authorized representative and will state the following:

"I certify under penalty of law that I personally have examined and am familiar with the waste through analysis and testing or through knowledge of the waste to support this certification that the waste complies with the treatment standards specified in 40 CFR Part 268 Subpart D and all applicable prohibitions set forth in 40 CFR 268.32 or RCRA Section 3004(d). I believe that the information I

submitted is true, accurate and complete. I am aware that there are significant penalties for submitting a false certification, including the possibility of a fine and imprisonment."

- If SCYI determines that a waste is subject to an exemption from land disposal prohibition, SCYI will notify the TSD facility in writing with each shipment that the waste is not prohibited from land disposal. At a minimum, the notification will include the EPA hazardous waste number, the manifest number and waste analysis data, if available.
- SCYI will maintain on-site a copy of all notices, certifications, demonstrations, waste analysis data, documentation supporting determinations based on knowledge of the waste and other information required under 40 CFR Part 268 for a period of five years from the date that the waste was last sent to an off-site TSD. This five year period may be extended in accordance with 40 CFR 268.7(a)(7).

TABLE 1

**Identification/EPA Classification of Hazardous Waste Stored
at the Hazardous Waste Storage Area**

Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico

WASTE GENERATED ¹	PROCESS GENERATING THE WASTE	BASIS FOR HAZARD CLASSIFICATION	EPA WASTE CODE	HAZARDOUS PROPERTIES OF WASTE
Process Waste	Petroleum Refining	Process Knowledge/Sampling Results	F037 F038 K049 K050 K051 K169 K171 K172 D001 D018	Toxic Ignitable
Commercial Product	Quality Assurance Testing	Knowledge of Waste	U154	Ignitable

1. Other hazardous waste, which may be generated periodically in limited quantities and which may include laboratory waste, spill residues, excess or off-specification raw materials or products, etc., will be stored at the hazardous waste storage area for periods less than 90 days.

TABLE 2**Analytical Parameters and Rationale**

Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico

WASTE ¹	WASTE PARAMETERS	RATIONALE FOR SELECTION
Process Waste ²	Volatile Organic Compounds (TCLP) Semi-Volatile Organic Compounds (TCLP) Metals (TCLP)	Ensure continued compliance with applicable regulations; Determine applicable requirements for off-site disposal
Ignitable	Flash Point	Ensure safe management of waste; Determine applicable requirements to treat, deactivate or separately manage ignitable wastes
Reactive	Releasable Cyanides Releasable Sulfides	Ensure safe management of waste; Determine applicable requirements to treat, deactivate or separately manage reactive wastes
Toxic (Benzene)	Benzene (TCLP)	Ensure continued compliance with applicable regulations; Determine applicable requirements for off-site disposal
Accumulated Liquid (HWSA Containment System)	Flash Point Benzene (TCLP)	Ensure proper classification as hazardous or non-hazardous. Determine applicable requirements for off-site disposal
Unknown Waste	pH Flash Point Releasable Cyanides Releasable Sulfides Volatile Organic Compounds (TCLP) Semi-Volatile Organic Compounds (TCLP) Metals (TCLP)	Ensure continued compliance with applicable regulations; Determine applicable requirements for off-site disposal

1. If waste is classified as hazardous based on knowledge, such as U154 or laboratory waste, sampling and analysis will not be conducted.
2. Includes F037, F038, K049, K050, K051, K169, K171, K172 wastes.

TABLE 3

Sampling Methods, Equipment, and Procedures

Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico

WASTE DESCRIPTION	SAMPLING METHOD	SAMPLING EQUIPMENT	SAMPLE PRESERVATION AND STORAGE
Process Sludges/Solids ¹	Sample collection method provided in Attachment A - SOP 001	Scoop Sampler	Preservation and storage procedures are based on analytical parameter; See Attachment A - SOP 001
Liquid Wastes ²	Sample collection method provided in Attachment A - SOP 002	Polyethylene Coliwasa	Preservation and storage procedures are based on analytical parameter; See Attachment A - SOP 002
Accumulated Liquids (HWSA Containment System)	S002 - Dipper (SW-846)	Disposable Beaker	Refer to Section 6.1 of SOP 002 for preservation and storage procedures based on analytical parameter.

1. Includes F037, F038, K049, K050, K051, K169, K171, K172, D001, D018 and other uncharacterized sludges and solids.

2. Includes characterized and uncharacterized liquid wastes.

TABLE 4

Sample Preparation and Analytical Methods

Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico

WASTE PARAMETER	SAMPLE EXTRACTION/ PREPARATION METHOD ⁽¹⁾	ANALYTICAL METHOD ⁽¹⁾
pH	N/A	9040B
Flash Point	N/A	1010
Releasable Cyanides	N/A	Section 7.3.3.2
Releasable Sulfides	N/A	Section 7.3.4.2
Volatile Organic Compounds	1311	8260B
Semi-Volatile Organic Compounds	1311	8270C
Arsenic	1311	6010B/6020/7060 ⁽³⁾
Barium	1311	6010B/6020/7080 ⁽³⁾
Cadmium	1311	6010B/6020/7130/7131A ⁽³⁾
Chromium	1311	6010B/6020/7190/7191 ⁽³⁾
Lead	1311	6010B/6020/7420/7421 ⁽³⁾
Mercury	1311	7470A/7471A ⁽³⁾
Selenium	1311	6010B/6020/7740/7741A/7742 ⁽³⁾
Silver	1311	6010B/6020/7760A/7761 ⁽³⁾

(1) Unless otherwise specified, all methods are described in "Test Methods For Evaluating Solid Waste: Physical/Chemical Methods", SW-846, Third Edition, 1986. Appropriate analytical methods will be selected based on the sample matrix, interferences, detection limits, etc.

(2) American Society for Testing and Materials, Philadelphia, Pa., "Annual Book of ASTM Standards".

(3) SW-846 Method 6010B will be used unless a lower method detection limit is required.

TABLE 5**Sampling Frequency for Waste Re-evaluation**

Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico

WASTE ¹	WASTE PARAMETERS	FREQUENCY
Process Waste ²	Volatile Organic Compounds (TCLP) Semi-Volatile Organic Compounds (TCLP) Metals (TCLP)	(3)
Ignitable	Flash Point	(3)
Reactive	Releasable Cyanides Releasable Sulfides	(3)
Toxic (Benzene)	Benzene (TCLP)	(3)
Accumulated Liquid (HWSA Containment System)	Flash Point Benzene (TCLP)	Daily (4)
Unknown Waste	pH Flash Point Releasable Cyanides Releasable Sulfides Volatile Organic Compounds (TCLP) Semi-Volatile Organic Compounds (TCLP) Metals (TCLP)	Every Batch

1. If waste is classified as hazardous based on knowledge, such as U154 or laboratory waste, sampling and analysis will not be conducted.
2. Includes F037, F038, K049, K050, K051, K169, K171, K172 wastes.
3. Sampling and analysis will be performed based on the requirements of the off-site TSDF used to treat and/or dispose of the hazardous wastes, or at times when process changes may significantly impact the characteristics of the hazardous waste generated and stored at the hazardous waste storage area.
4. Sample collection will be performed upon discovery of standing liquids if containers storing free liquids are present in the HWSA.

TABLE 6

Land Disposal Restriction (LDR) Treatment Standards

Shell Chemical Yabucoa, Inc.
Yabucoa, Puerto Rico

WASTE	PARAMETERS	WASTEWATER (mg/L)	NON- WASTEWATER (mg/Kg)
Process Waste ¹	Acenaphthene	0.059	3.4 (NA for K051)
	Benzene	0.14	10
	Benz(a)anthracene	0.059	3.4
	Benzo(g,h,i)perylene (K169 only)	0.0055	1.8
	Benzo(a)pyrene	0.061	3.4
	bis(2-Ethylhexyl)phthalate	0.28	28
	Carbon disulfide (K049 only)	3.8	NA
	Chrysene	0.059	3.4
	2,4-Dimethylphenol (K049 only)	0.036	NA
	Di-n-butyl phthalate	0.057	28
	Ethylbenzene	0.057	10
	Fluorene	0.059	NA (3.4 for K169)
	Naphthalene	0.059	5.6
	Phenanthrene	0.059	5.6
	Phenol	0.039	6.2
	Pyrene	0.067	8.2
	Toluene	0.080	10
	Xylenes	0.32	30
	Cyanides (Total)	1.2	590
	Antimony (K172 only)	1.9	1.15 mg/L TCLP
	Arsenic (K171 & K172 only)	1.4	5.0 mg/L TCLP
	Chromium (Total)	2.77	0.6 mg/L TCLP
	Lead	0.69	0.75 mg/L TCLP
	Nickel	NA (3.98 for K171 & K172)	11 mg/L TCLP
	Vanadium (K171 & K172 only)	4.3	1.6 mg/L TCLP

1. Includes F037, F038, K049, K050, K051, K169, K171, K172 wastes.

WASTE	PARAMETERS	WASTEWATER (mg/L)	NON- WASTEWATER (mg/Kg)
Ignitable	----	Deactivation	Deactivation ⁽¹⁾
Reactive	Cyanides (Total)	---	590
	Cyanides (Amenable)	0.86	30
	Sulfides (K172 only)	Deactivation	Deactivation
Toxic (Benzene)	Benzene (TCLP)	0.14	10 ⁽¹⁾

(1) In addition, all underlying hazardous constituents must meet Universal Treatment Standards found at 40 CFR 268.48.

Figure 1

Waste Compatibility Matrix

Figure 1

Waste Compatibility Matrix

Reactivity group no.	Reactivity group name	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	101	102	103	104	105	106	107	
1	Acids, mineral, nonoxidizing																																										
2	Acids, mineral, oxidizing																																										
3	Acids, organic																																										
4	Alcohols and glycols																																										
5	Aldehydes																																										
6	Amines																																										
7	Amines, aliphatic and aromatic																																										
8	Azo compounds, diazo compounds, and hydrazines																																										
9	Carbonates																																										
10	Ceramics																																										
11	Cyanides																																										
12	Dithiocarbamates																																										
13	Esters																																										
14	Ethers																																										
15	Fluorides, inorganic																																										
16	Hydrocarbons, aromatic																																										
17	Halogenated organics																																										
18	Heavy metals																																										
19	Ketones																																										
20	Mercaptans and other organic sulfides																																										
21	Metals, solid and soluble earth, elemental																																										
22	Metals, other elemental & alloys as powders, vapors, or sponges																																										
23	Metals, other elemental & alloys as sheets, rods, drops, moldings, etc.																																										
24	Metals and metal compounds, toxic																																										
25	Nitrides																																										
26	Nitroes																																										
27	Nitro compounds, organic																																										
28	Hydrocarbons, aliphatic, unsaturated																																										
29	Hydrocarbons, aliphatic, saturated																																										
30	Peroxides and hydroperoxides, organic																																										
31	Phenols and cresols																																										
32	Organophosphates, phosphonates, phosphorothioates																																										
33	Sulfides, inorganic																																										
34	Epoxides																																										
101	Combustible and flammable materials, miscellaneous																																										
102	Explosives																																										
103	Polymerizable compounds																																										
104	Quenching agents, strong																																										
105	Reducing agents, strong																																										
106	Water and mixtures containing water																																										
107	Water reactive substances																																										

Reactivity Code

H	F	G	GT	GF	E	P	S	U
---	---	---	----	----	---	---	---	---

Consequences

Heat generation
Fire
Innocuous and nonflammable gas
Toxic gas generation
Flammable gas generation
Explosion
Violent polymerization
Solubilization of toxic substances
May be hazardous but unknown

Example:

H	F	GT
---	---	----

Heat generation, fire, and toxic gas generation

DO NOT MIX WITH ANY CHEMICAL OR WASTE MATERIAL

EXTREMELY REACTIVE!

EXTREMELY REACTIVE!

Reactivity Code

H

F

G

GT

GF

E

P

S

U

Consequences

Heat generation

Fire

Innocuous and nonflammable gas generation

Toxic gas generation

Flammable gas generation

Explosion

Violent polymerization

Solubilization of toxic substances

May be hazardous but unknown

Example:

H

F

GT

Heat generation, fire, and toxic gas generation

ATTACHMENT A

Subject or Title:

Page 1 of 4

Sampling of Hazardous Sludges

SOP No. :

Revision No. :

Effective Date:

001

1.0 Introduction

The purpose of this Standard Operating Procedure (SOP) is to establish a protocol for sampling of hazardous sludges generated at the SCYI facility in order to ensure compliance with applicable Resource Conservation and Recovery Act (RCRA) regulations.

2.0 Sampling Method

- All sludge samples will be collected using a scoop as per ASTM D5633 and in accordance with procedures set forth in "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846.
- Prior to sampling, all sampling personnel will don appropriate personal protection equipment which includes, but not limited to, hard hats, coveralls (Tyvek Suits), gloves, safety goggles, steel toed boots, etc.

3.0 Type of Hazardous Waste Container**3.1 Drums**

- Sampling procedures presented in this SOP are applicable to 55 gallon drums, 30 gallon drums, and 1 cyd. sacks and bags.
- Open, sample and close each drum individually to minimize the risk of exposure.
- For drums with bungs, loosen the large bung slowly using non-sparking tools, whereas, for drums with removable lids, loosen the ring slowly with non-sparking manual wrench or air impact wrench.
- Don new pair of gloves prior to collecting each sample.
- All sampling equipment will be laboratory or factory-cleaned, or decontaminated in accordance with Section 5.0 of this SOP, prior to use.
- At a random location within the drum, scrap and clear the top 1 inch of the sludge with appropriate equipment. Collect a grab sample from the cleared location using a stainless steel scoop at depth no greater than 6 inches from the undisturbed sludge surface. Extraction of samples might require tilting of the container.
- Transfer the sample into a sample bottle/vial with the aid of a spatula.
- Follow the procedures provided in Section 6.1 for preservation and transport.

Subject or Title:

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001

Revision No. :

Effective Date:

3.2 Roll-off Bins

- Open, sample and close each bin individually to minimize risk of exposure. Opening and closing of the covers on the bins will be conducted by a trained personnel using non-sparking equipment.
- Don new pair of gloves prior to collecting each sample.
- All sampling equipment will be laboratory or factory-cleaned, or decontaminated in accordance with Section 5.0 of this SOP, prior to use.
- For each bin sampled, at a random location within the bin, scrap and clear the top 1 inch of the sludge with appropriate equipment. Collect a grab sample from the cleared location using a stainless steel scoop at depth no greater than 6 inches from the undisturbed sludge surface.
- Transfer the sample into a sample bottle/vial with the aid of a spatula.
- Follow the procedures provided in Section 6.1 for preservation and transport.

4.0 Number of Samples**4.1 Homogeneous**

- If the sludge contained in a group of containers (drums, sacks or bags, bins) is from a single source, (e.g., from a single tank or a single spill location), one sample (individual or composite based on the analytical parameter) will be collected from a single container (drum, sack or bag, bin) to be selected at random.

4.2 Heterogeneous

- If the sludge in a group of containers (drums, sacks or bags, bins) is from multiple or unknown sources, the following procedure (as per ASTM D140) will be followed to determine the number of containers (drums, sacks or bags, bins) to be sampled.
- Count the total number of containers (drums, bags or sacks, bins) in the batch to be sampled.
- Calculate the cube root of the total number of containers in the batch. The following table provides the number of containers to be samples for different population ranges.

Subject or Title:

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Sampling of Hazardous Sludges

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Total No. of Containers	No. of Containers to be Sampled
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5

- Select at random the number of containers (drums, bags or sacks, bins) to be sampled from the batch.
- Collect one sample from each container (drums, bags or sacks, bins) selected.

5.0 Decontamination Procedures

- Clean and decontaminate reusable sampling equipment as follows.
- Scrub all visual contamination from the field sampling equipment using a laboratory grade glassware detergent and tap water.
- Generously rinse with tap water.
- Finally, rinse with distilled and deionized water.
- Wrap the cleaned equipment with clean aluminum foil until use in the field.

6.0 QA/QC Procedures

6.1 Sample Collection and Handling

- Laboratory cleaned and appropriately labeled sampling containers will be supplied by the analytical laboratory, prior to the field sampling activity.
- Follow the table below for sample container, container volume, preservation and holding time for the samples collected as per this SOP.

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Sampling of Hazardous Sludges

SOP No. :
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Parameter	Sample Container	Container Volume	Preservation	Maximum Holding Time
Volatile Organic Compounds	Wide mouth Glass, Teflon liner cap	4 oz	Cool to 4°C	14 days
Semi-Volatile Organic Compounds	Wide mouth Glass, Teflon liner cap	8 oz	Cool to 4°C	14 days
Metals (except Cr VI and Hg)	Plastic or Glass	4 oz	Cool to 4°C	180 days
Hg	Plastic or Glass	4 oz	Cool to 4°C	28 days
Hazardous Characteristic	Plastic or Glass	8 oz	NA	NA

- Log the date, time, name of sampling personnel, sample ID, sampling matrix, analytical parameters, etc. in a field log book.
- Fill out a chain-of-custody form supplied by the laboratory analyzing the samples. A sample chain-of-custody form is attached.

6.2 QA/QC Samples

- Duplicates - For drums, bags or sacks, and bins, collect a duplicate sample for every 20 samples collected. Collect the duplicate sample from the same container (drums, bags or sacks, bins) as the original sample, but from a different location. Follow the procedure as per Section 3.0 for collecting duplicate samples.
- Field Blanks - Field blanks will be collected and analyzed for volatile organics if the field samples collected during a sampling event are analyzed for volatile organics. One field blanks will be collected for every 20 samples collected or for every sampling event if the number of samples collected is less than 20.
- Trip Blanks - Trip blanks are not required for sludge (non-aqueous matrix) sampling.

[illegible]

Subject or Title:

Page 1 of 4

Sampling of Hazardous Liquids

SOP No. :
002

Revision No. :

Effective Date:

1.0 Introduction

The purpose of this Standard Operating Procedure (SOP) is to establish a protocol for sampling of liquid hazardous waste generated at the SCYI facility in order to ensure compliance with applicable Resource Conservation and Recovery Act (RCRA) regulations.

2.0 Sampling Method

- All liquid hazardous waste samples will be collected using a disposable polyethylene Coliwas sampler as per ASTM D5495 and in accordance with procedures set forth in "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", SW-846.
- Prior to sampling, all sampling personnel will don appropriate personal protection equipment including, but not limited to, hard hats, coveralls (Tyvek Suits), gloves, safety goggles, steel toed boots, etc.

3.0 Type of Hazardous Waste Container**3.1 Drums**

- Sampling procedures presented in this SOP are applicable to 55 gallon drums, 30 gallon drums and other liquid waste storage containers.
- Open, sample and close each drum individually to minimize the risk of exposure.
- For drums with bungs, loosen the large bung slowly using non-sparking tools, whereas, for drums with removable lids, loosen the ring slowly with non-sparking manual wrench or air impact wrench.
- Don new pair of gloves prior to collecting each sample.
- All sampling equipment will be laboratory or factory-cleaned, or decontaminated in accordance with Section 5.0 of this SOP, prior to use.
- With the sampler in the open position, collect a sample by vertically inserting the Coliwas into the liquid waste drum or container. The sample is collected from a depth midway between the top and the bottom of the drum.
- Collect the sample at the desired depth by rotating the handle until one leg of the T is squarely perpendicular against the locking block.

Subject or Title:

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Sampling of Hazardous Liquids

SOP No. :
002

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- Upon sample collection, rotate the handle back to the closed position by turning the handle until one leg of the T is parallel to the locking block.
- Withdraw the sampler and transfer the sample into the sample bottle/vial. Use a funnel to avoid spillage if necessary.
- Follow the procedures provided in Section 6.1 for preservation and transport.
- Dispose of the sampler as a hazardous waste.

4.0 Number of Samples**4.1 Homogeneous Wastes**

- If the waste liquid contained in a group of drums or other containers is from a single source, (e.g., from a single tank or a single spill location), one grab sample will be collected from a single drum or container to be selected at random.

4.2 Heterogeneous Wastes

- If the waste liquid in a group of drums or other containers is from multiple or unknown sources, the following procedure (as per ASTM D140) will be followed to determine the number of drums or containers to be sampled.
- Count the total number of drums or containers in the batch to be sampled.
- Calculate the cube root of the total number of drums or containers in the batch. The following table provides the number of drums or containers to be sampled for different population ranges.

Total No. of Drums or Containers	No. of Drums or Containers to be Sampled
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5

Subject or Title:

Page 3 of 4

Sampling of Hazardous Liquids

SOP No. :
002

Revision No. :

Effective Date:

- Select at random the number of drums or containers to be sampled from the batch.
- Collect one grab sample from each drum or container selected.

5.0 Decontamination Procedures

- Clean and decontaminate reusable sampling equipment as follows.
- Scrub all visual contamination from the field sampling equipment using a laboratory grade glassware detergent and tap water.
- Generously rinse with tap water.
- Rinse with distilled and deionized water.
- If sample is to be analyzed for metals, rinse the equipment with 10% nitric acid (trace metal or higher grade) diluted with distilled and deionized water. Rinse with distilled and deionized water.
- If sample is to be analyzed for organics, rinse the equipment with Acetone (pesticide grade) and air dry. Rinse with distilled and deionized water.
- Wrap the cleaned equipment with clean aluminum foil until use in the field.

6.0 QA/QC Procedures

6.1 Sample Collection and Handling

- Laboratory-cleaned and appropriately labeled sampling containers will be supplied by the analytical laboratory prior to the field sampling activity.
- Follow the table below for sample container, container volume, preservation and holding time for the samples collected as per this SOP.

Subject or Title:

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Sampling of Hazardous Liquids

SOP No. :
002

Revision No. :

Effective Date:

Parameter	Sample Container	Container Volume	Preservation	Maximum Holding Time
Volatile Organic Compounds	Glass vial, Teflon lined septum cap	40 ml	4 drops of conc. HCl, Cool to 4°C	14 days
Semi-Volatile Organic Compounds	Amber Glass, Teflon liner cap	1 gallon	Cool to 4°C	Extraction 7 days; Analysis 40 days from extraction
Metals (except Cr VI and Hg)	Plastic or Glass	600 ml	Add HNO ₃ to pH < 2, Cool to 4°C	180 days
Hg	Plastic or Glass	400 ml	Add HNO ₃ to pH < 2, Cool to 4°C	28 days
Hazardous Characteristic	Plastic or Glass	500 ml	NA	NA

- Log the date, time, name of sampling personnel, sample ID, sampling matrix, analytical parameters, etc. in a field log book.
- Fill out a chain-of-custody form supplied by the laboratory analyzing the samples. A sample chain-of-custody form is attached.

6.2 QA/QC Samples

- Duplicates - For drums or containers, collect a duplicate sample for every 20 samples collected. Follow the procedure as per Section 3.0 for collecting duplicate samples. Collect the duplicate with original sample by alternately filling the sample containers from the same sampling device.
- Field Blanks - Field blanks will be collected and analyzed for the same parameters as the field samples. One field blank will be collected for every 20 samples collected or for every sampling event if the number of samples collected is less than 20.
- Trip Blanks - Trip blanks are required for liquid waste (aqueous matrix) sampling. The trip blanks consist of sample containers filled with laboratory-demonstrated analyte free water. These samples accompany the bottles that are prepared at the lab into the field and back to the laboratory along with the collected samples for analysis. Trip blanks will be analyzed for volatile organics. One trip blank will be included per sample shipment.

[illegible]

Appendix Q-1

Hazardous Wastes Laboratory Analysis

PUERTO RICO SUN OIL CORPORATION
PO BOX 186
YABUCOA, PR 00767

ATTN: MRS. JULIE ROSADO

DS# 99-0371
Sample collected by Client
Limit = Maximum Contaminant Level

Date: August 26, 1999
Work Order #: 147-01-05
EQ Lab Sample #: 99-07D24
Date Sample collected: 07/30/99
Date Sample received: 07/30/99
Source: Catalyst 324
Description: Solid - Grab

SAMPLE IDENTIFICATION

<u>Sample Number</u>	<u>Sample Description</u>	<u>Sample Number</u>	<u>Sample Description</u>
1	Catalyst 324		



A 604163

Order # 99-07-D24
08/26/99

Page 2

TEST RESULTS BY SAMPLE

Sample: 01A Catalyst 324
Job: 02-04 Solid - Grab

Collected: 07/30/99

<u>Test Description</u>	<u>Result</u>	<u>Limit</u>	<u>Units</u>	<u>Analyzed</u>	<u>By</u>
TCLP EXTRACTION					
Zero Headspace Ext.			on	08/02/99	JFD
Semi-Vol. & Metals Ext.			on	08/02/99	HC
TCLP METALS					
Total Arsenic	0.027	5.0	mg/L	08/03/99	GR
Total Barium	<0.1	100	mg/L	08/03/99	GR
Total Cadmium	<0.002	1.0	mg/L	08/03/99	GR
Total Chromium	0.051	5.0	mg/L	08/03/99	GR
Total Lead	<0.005	5.0	mg/L	08/03/99	GR
Total Mercury	<0.002	0.20	mg/L	08/04/99	JC
Total Selenium	<0.001	1.0	mg/L	08/03/99	GR
Total Silver	0.020	5.0	mg/L	08/03/99	GR
CORROSIVITY					
pH	10.16	2.0-12.5	S.U.	07/30/99	FMR
IGNITABILITY					
	Does not ignite			08/04/99	FMR
REACTIVITY					
Releasable Cyanide	<1.0	250	mg/kg	08/06/99	LS
Releasable Sulfide	<1.0	500	mg/kg	08/02/99	MG
TCLP VOLATILES					
Benzene	<0.10	0.50	mg/L	08/03/99	IPM
Carbon Tetrachloride	<0.10	0.50	mg/L	08/03/99	IPM
Chlorobenzene	<0.10	100	mg/L	08/03/99	IPM
Chloroform	<0.10	6.0	mg/L	08/03/99	IPM
1,4-Dichlorobenzene	<0.10	7.5	mg/L	08/03/99	IPM
1,2-Dichloroethane	<0.10	0.50	mg/L	08/03/99	IPM
1,1-Dichloroethene	<0.10	0.70	mg/L	08/03/99	IPM
Tetrachloroethene	<0.10	0.70	mg/L	08/03/99	IPM
Trichloroethene	<0.10	0.50	mg/L	08/03/99	IPM
Vinyl Chloride	<0.10	0.20	mg/L	08/03/99	IPM
Methyl Ethyl Ketone	<1.0	200	mg/L	08/03/99	IPM
TCLP SEMI-VOLATILES					
Pyridine	<5.0	5.0	mg/L	08/08/99	ASA
Nitrobenzene	<0.1	2.0	mg/L	08/08/99	ASA
2,4-Dinitrotoluene	<0.1	0.13	mg/L	08/08/99	ASA
Hexachlorobenzene	<0.001	0.13	mg/L	08/08/99	ASA
Hexachloro-1,3-Butadiene	<0.01	0.50	mg/L	08/08/99	ASA
Hexachloroethane	<0.01	3.0	mg/L	08/08/99	ASA

Order # 99-07-D24
08/26/99

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TEST RESULTS BY SAMPLE

<u>Test Description</u>	<u>Result</u>	<u>Limit</u>	<u>Units</u>	<u>Analyzed</u>	<u>By</u>
Pentachlorophenol	<0.1	100	mg/L	08/08/99	ASA
2,4,6-Trichlorophenol	<1.0	400	mg/L	08/08/99	ASA
2,4,6-Trichlorophenol	<1.0	2.0	mg/L	08/08/99	ASA
o-Cresol - Note #1	<1.0	200	mg/L	08/08/99	ASA
m,p-Cresol - Note #1	<1.0	200	mg/L	08/08/99	ASA
Extraction Date			on	08/03/99	HC

TCLP PESTICIDES

Chlordane	<0.003	0.030	mg/L	08/08/99	JOA
Methoxychlor	<0.010	10	mg/L	08/08/99	JOA
Toxaphene	<0.050	0.50	mg/L	08/08/99	JOA
Lindane	<0.005	0.40	mg/L	08/08/99	JOA
Heptachlor	<0.005	0.0080	mg/L	08/08/99	JOA
Heptachlor Epoxide	<0.005	0.0080	mg/L	08/08/99	JOA
Endrin	<0.005	0.020	mg/L	08/08/99	JOA
Extraction Date			on	08/03/99	HC

TCLP HERBICIDES

2,4,5-TP (Silvex)	<0.010	1.0	mg/L	08/08/99	JOA
2,4-D	<0.100	10	mg/L	08/08/99	JOA
Extraction Date			on	08/03/99	JFD

Note:

#1) Where cresols cannot be differentiated regulatory level for Total Cresol is 200 mg/L.

PUERTO RICO SUN OIL CORPORATION
P.O. BOX 186
YABUCOA, P.R. 00767

Attn: MRS. JULIE ROSADO

Order #: 17430
Date: March 01, 2000
Work ID: Solid - Grab
Date Received: 02/24/00
Date Completed: 03/01/00
Client Code: 147-01

DS#: 99-11465
Sample collected by client
Limit = Maximum Contaminant Level

SAMPLE IDENTIFICATION

Sample
Number
01

Sample
Description
Katalco 46-1, 46.46-4

Sample
Number

Sample
Description



ENVIRONMENTAL QUALITY LABORATORIES, INC.

P.O. BOX 11458, SAN JUAN, P.R. 00910-1458 • TEL: (787) 725-5333 • FAX (787) 724-3110



Order # : 17430

03/01/00

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TEST RESULTS BY SAMPLE

Sample: 01A Katalco 46-1, 46-4
Job: 01- 05 Solid - Grab

Collected: 02/23/00

<u>Test Description</u>	<u>Result</u>	<u>Limit</u>	<u>Units</u>	<u>Analyzed</u>	<u>By</u>
TCLP EXTRACTION					
Zero Headspace Ext.			on	02/25/00	CIL
Semi-Vol. & Metals Ext.			on	02/25/00	CIL
TCLP METALS					
Total Arsenic	<0.003	5.0	mg/L	02/28/00	GR
Total Barium	<0.1	100	mg/L	02/28/00	GR
Total Cadmium	<0.002	1.0	mg/L	02/28/00	GR
Total Chromium	<0.002	5.0	mg/L	02/28/00	GR
Total Lead	<0.005	5.0	mg/L	02/28/00	GR
Total Mercury	<0.002	0.20	mg/L	02/28/00	GR
Total Selenium	<0.001	1.0	mg/L	03/01/00	JC
Total Silver	<0.001	5.0	mg/L	02/28/00	GR
CORROSIVITY					
pH	10.85	2.0-12.5	S.U.	02/25/00	FMR
IGNITABILITY	Does not ignite			03/01/00	FMR
REACTIVITY					
Releasable Cyanide	<1.0	250	mg/kg	02/29/00	KV
Releasable Sulfide	<1.0	500	mg/kg	02/28/00	MG
TCLP VOLATILES					
Benzene	<0.10	0.50	mg/L	02/26/00	IPM
Carbon Tetrachloride	<0.10	0.50	mg/L	02/26/00	IPM
Chlorobenzene	<0.10	100	mg/L	02/26/00	IPM
Chloroform	<0.10	6.0	mg/L	02/26/00	IPM
1,4-Dichlorobenzene	<0.10	7.5	mg/L	02/26/00	IPM
1,2-Dichloroethane	<0.10	0.50	mg/L	02/26/00	IPM
1,1-Dichloroethene	<0.10	0.70	mg/L	02/26/00	IPM
Tetrachloroethene	<0.10	0.70	mg/L	02/26/00	IPM
Trichloroethene	<0.10	0.50	mg/L	02/26/00	IPM
Vinyl Chloride	<0.10	0.20	mg/L	02/26/00	IPM
Methyl Ethyl Ketone	<1.00	200	mg/L	02/26/00	IPM



ENVIRONMENTAL QUALITY LABORATORIES, INC.

P.O. BOX 11456, SAN JUAN, P.R. 00910-1456 • TEL: (787) 725-5333 • FAX (787) 724-3110



Order #: 17430
03/01/00

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TEST RESULTS BY SAMPLE

Sample: 01A Katalco 46-1, 46-4
Job: 01- 05 Solid - Grab

Collected: 02/23/00

<u>Test Description</u>	<u>Result</u>	<u>Limit</u>	<u>Units</u>	<u>Analyzed</u>	<u>By</u>
TCLP SEMI-VOLATILES					
Pyridine	<1.0	5.0	mg/L	02/28/00	ASA
Nitrobenzene	<1.5	2.0	mg/L	02/28/00	ASA
2,4-Dinitrotoluene	<0.10	0.13	mg/L	02/28/00	ASA
Hexachlorobenzene	<0.01	0.13	mg/L	02/28/00	ASA
Hexachloro-1,3-Butadiene	<0.25	0.50	mg/L	02/28/00	ASA
Hexachloroethane	<0.20	3.0	mg/L	02/28/00	ASA
Pentachlorophenol	<3.0	100	mg/L	02/28/00	ASA
2,4,5-Trichlorophenol	<0.05	400	mg/L	02/28/00	ASA
2,4,6-Trichlorophenol	<0.5	2.0	mg/L	02/28/00	ASA
o-Cresol - Note #1	<1.0	200	mg/L	02/28/00	ASA
m,p-Cresol - Note #1	<1.0	200	mg/L	02/28/00	ASA
Extraction Date			on	02/26/00	CIL
TCLP PESTICIDES					
Chlordane	<0.003	0.030	mg/L	03/01/00	JOA
Methoxychlor	<0.010	10	mg/L	03/01/00	JOA
Toxaphene	<0.050	0.50	mg/L	03/01/00	JOA
Lindane	<0.005	0.40	mg/L	03/01/00	JOA
Heptachlor	<0.005	0.0080	mg/L	03/01/00	JOA
Heptachlor Epoxide	<0.005	0.0080	mg/L	03/01/00	JOA
Endrin	<0.005	0.020	mg/L	03/01/00	JOA
Extraction Date			on	02/26/00	CIL
TCLP HERBICIDES					
2,4,5-TP (Silvex)	<0.010	1.0	mg/L	03/01/00	JOA
2,4-D	<0.100	10	mg/L	03/01/00	JOA
Extraction Date			on	02/26/00	CIL

Note:

#1) Where cresols cannot be differentiated regulatory level for Total Cresol is 200 mg/L.



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P.O. BOX 11458, SAN JUAN, P.R. 00910-1458 • TEL: (787) 725-5333 • FAX (787) 724-3110



.....
Drying
BEDS - Biological
TCLP

PUERTO RICO SUN OIL CORPORATION
P.O. BOX 186
YABUCOA, P.R. 00767

Attn: MRS. JULIE ROSADO

Order #: 8873

Date: February 01, 00

Work ID: Solid - Grab

Date Received: 01/26/00

Date Completed: 02/01/00

Client Code: 147-01

DS#: 99-11463

Sample collected by client

Limit = Maximum Contaminant Level

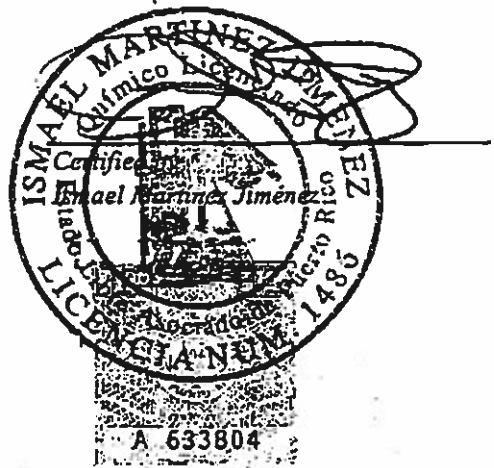
SAMPLE IDENTIFICATION

Sample
Number
→ 1

Sample
Description
Sand Drying Beds - BIOLOGICAL
SLUDGE

Sample
Number

Sample
Description



ENVIRONMENTAL QUALITY LABORATORIES, INC.

P.O. BOX 11458, SAN JUAN, P.R. 00910-1458 • TEL: (787) 725-5333 • FAX (787) 724-3110



Order # : 8873
02/01/00

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TEST RESULTS BY SAMPLEBIOLOGICAL SLUDGE

Sample: 01A Sand Drying Beds
Job: 01-05 Solid - Grab

Collected: 01/26/00

<u>Test Description</u>	<u>Result</u>	<u>Limit</u>	<u>Units</u>	<u>Analyzed</u>	<u>By</u>
TCLP EXTRACTION					
Zero Headspace Ext.			on	01/28/00	HC
Semi-Vol. & Metals Ext.			on	01/27/00	HC
TCLP METALS					
Total Arsenic	<0.003	5.0	mg/L	01/31/00	JAG
Total Barium	<0.1	100	mg/L	01/31/00	JAG
Total Cadmium	<0.002	1.0	mg/L	01/31/00	JAG
Total Chromium	<0.002	5.0	mg/L	01/31/00	JAG
Total Lead	<0.005	5.0	mg/L	01/31/00	JAG
Total Mercury	<0.002	0.20	mg/L	02/01/00	JC
Total Selenium	<0.001	1.0	mg/L	01/31/00	JAG
Total Silver	<0.001	5.0	mg/L	01/31/00	JAG
CORROSIVITY					
pH	7.18	2.0-12.5	S.U.	01/27/00	FMR
IGNITABILITY	Does not ignite			01/27/00	FMR
REACTIVITY					
Releasable Cyanide	<1.0	250	mg/kg	02/01/00	LS
Releasable Sulfide	12.4	500	mg/kg	01/28/00	MG
TCLP VOLATILES					
Benzene	<0.10	0.50	mg/L	01/29/00	JDA
Carbon Tetrachloride	<0.10	0.50	mg/L	01/29/00	JDA
Chlorobenzene	<0.10	100	mg/L	01/29/00	JDA
Chloroform	<0.10	6.0	mg/L	01/29/00	JDA
1,4-Dichlorobenzene	<0.10	7.5	mg/L	01/29/00	JDA
1,2-Dichloroethane	<0.10	0.50	mg/L	01/29/00	JDA
1,1-Dichloroethene	<0.10	0.70	mg/L	01/29/00	JDA
Tetrachloroethene	<0.10	0.70	mg/L	01/29/00	JDA
Trichloroethene	<0.10	0.50	mg/L	01/29/00	JDA
Vinyl Chloride	<0.10	0.20	mg/L	01/29/00	JDA
Methyl Ethyl Ketone	<1.00	200	mg/L	01/29/00	JDA



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Order #: 8873

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02/01/00

TEST RESULTS BY SAMPLEBIOLOGICAL SLUDGE

Collected: 01/26/00

Sample: 01A Sand Drying Beds
Job: 01- 05 Solid - Grab

<u>Test Description</u>	<u>Result</u>	<u>Limit</u>	<u>Units</u>	<u>Analyzed</u>	<u>By</u>
TCLP SEMI-VOLATILES					
Pyridine	<1.0	5.0	mg/L	01/31/00	ASA
Nitrobenzene	<1.5	2.0	mg/L	01/31/00	ASA
2,4-Dinitrotoluene	<0.10	0.13	mg/L	01/31/00	ASA
Hexachlorobenzene	<0.01	0.13	mg/L	01/31/00	ASA
Hexachloro-1,3-Butadiene	<0.25	0.50	mg/L	01/31/00	ASA
Hexachloroethane	<0.20	3.0	mg/L	01/31/00	ASA
Pentachlorophenol	<3.0	100	mg/L	01/31/00	ASA
2,4,5-Trichlorophenol	<0.05	400	mg/L	01/31/00	ASA
2,4,6-Trichlorophenol	<0.5	2.0	mg/L	01/31/00	ASA
o-Cresol - Note #1	<1.0	200	mg/L	01/31/00	ASA
m,p-Cresol - Note #1	<1.0	200	mg/L	01/31/00	ASA
Extraction Date			on	01/28/00	CIC
TCLP PESTICIDES					
Chlordane	<0.003	0.030	mg/L	01/29/00	JOA
Methoxychlor	<0.010	10	mg/L	01/29/00	JOA
Toxaphene	<0.050	0.50	mg/L	01/29/00	JOA
Lindane	<0.005	0.40	mg/L	01/29/00	JOA
Heptachlor	<0.005	0.0080	mg/L	01/29/00	JOA
Heptachlor Epoxide	<0.005	0.0080	mg/L	01/29/00	JOA
Endrin	<0.005	0.020	mg/L	01/29/00	JOA
Extraction Date			on	01/28/00	CIC
TCLP HERBICIDES					
2,4,5-TP (Silvex)	<0.010	1.0	mg/L	01/29/00	JOA
2,4-D	<0.100	10	mg/L	01/29/00	JOA
Extraction Date			on	01/28/00	CIC

Note:

#1) Where cresols cannot be differentiated regulatory level for Total Cresol is 200 mg/L.



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